VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF GAMMA ISOTOPES

(GL-RAD-A-013 REVISION 25)

APPLICABLE TO METHODS:
EPA 600/4-80-032 Method 901.1
DOE EML HASL-300 Section 4.5.2.3
DOE EML HASL-300 Ga-01-R

PROPRIETARY INFORMATION

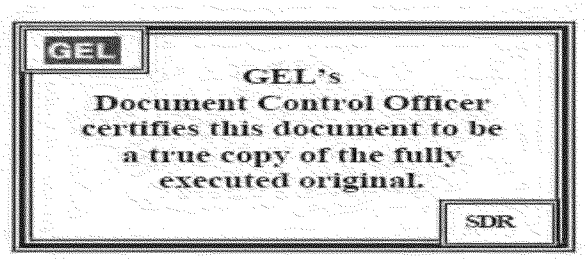

TABLE OF CONTENTS

1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF GAMMA ISOTOPES....3
2.0 METHOD OBJECTIVE, PURPOSE, AND SUMMARY....3
3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT....3
4.0 METHOD VARIATIONS....4
5.0 DEFINITIONS....4
6.0 INTERFERENCES....4
7.0 SAFETY PRECAUTIONS AND WARNINGS....4
8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION....5
9.0 REAGENTS, CHEMICALS, AND STANDARDS....5
10.0 SAMPLE HANDLING AND PRESERVATION....6
11.0 SAMPLE PREPARATION....6
12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS....8
13.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE....8
14.0 ANALYSIS AND INSTRUMENT OPERATION....8
15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE....8
16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS....8
17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL....9
18.0 RECORDS MANAGEMENT....9
19.0 LABORATORY WASTE HANDLING AND DISPOSAL....9
20.0 REFERENCES....9
21.0 HISTORY....10

1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF GAMMA ISOTOPES

2.0 METHOD OBJECTIVE, PURPOSE, AND SUMMARY

2.1 This standard operating procedure (SOP) provides the necessary instructions to conduct the analysis for gamma isotopes in water, soil, urine, filters, drinking water and miscellaneous matrices.

2.2 Water samples are typically counted in Marinelli beakers. Soil samples are typically sealed in aluminum cans, which can be counted immediately if Ra-226 is not desired. If Ra-226 is desired, the sealed can is set aside for minimum of 20 days to allow equilibrium between Rn-222 and Bi-214 to become re-established. Ra-226 is then quantified using the 609 keV line of Bi-214.

2.3 This method is based on the source method EPA 600/4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," August 1980, Method 901.1, and the Department of Energy (DOE) EML Procedures Manual source method for Gamma PHA in environmental samples, HASL-300 Section 4.5.2.3 and Ga-01-R, Gamma Radioassay.

2.4 This SOP is applicable for analyzing samples that contain radionuclides emitting gamma photons with energies ranging from about 5 to 2000 keV (including I-131).

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

3.1 Minimum Detectable Activity (MDA): The MDA is based upon sample volume, Compton background, instrument efficiency, count time, and other statistical factors, as well as specific isotopic values such as abundance and half-life. A typical detection limit is 10 pCi/L or 0.1 pCi/g (based on Cs-137). The MDA for drinking water samples is 10 pCi/L (based on Cs-137).

3.2 Method Precision: Typical Relative Percent Difference (RPD) is 20% or less or 100% or less if the activity is less than five times the MDA.

3.3 Method Bias (Accuracy): The method accuracy requirement for gamma, measured by running a Laboratory Control Sample (LCS) with each batch, is 25% of the true value. For drinking water samples, laboratory fortified blanks (LFB, equivalent to LCS) recoveries should be between 90-110% of the known value.

3.4 Procedures contained in this SOP may be used to analyze REMP samples.

3.5 Analysts training records are maintained as quality records as outlined in GL-QS-E-008. Analysts training and proficiency in the method is outlined in the Employee Training SOP GL-HR-E-002.

3.6 For drinking water samples, analyst initial and ongoing demonstrations of proficiency will follow critical elements for radiochemisrtry, chapter VI, section 1.5, of The Manual for the Certification of Laboratories Analyzing Drinking Water (reference 20.5).

3.7 Sensitivity studies will follow critical elements for radiochemistry, chapter VI, section 7.3 of The Manual for the Certification of Laboratories Analyzing Drinking Water (reference 20.5).

4.0 METHOD VARIATIONS

4.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Team Leader. Variations to a method will be documented with the analytical raw data.

4.2 Filter samples can either be counted directly, or digested prior to counting. If filters are digested, they are digested in accordance with GL-RAD-A-026.

4.3 No method modifications are permitted for drinking water samples.

5.0 DEFINITIONS

5.1 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

5.2 Deionized (DI) water: Type I water.Refer to GL-LB-E-016.

5.3 AlphaLIMS: GEL's Laboratory Information Management System.

5.4 Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.

5.5 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.

5.6 Laboratory Duplicate (DUP): For soils, when sufficient sample is available, a separate duplicate will be prepared. For liquid samples and when sufficient sample is not available for solids, an independent count of the sample container will be performed to show precision.

5.7 Laboratory Control Sample (LCS): A sample matrix, similar to the batch of associated samples (when available) that is free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. The LCS is equivalent to a Fortified Blank in the EPA drinking water compliance manual (See to section 20.5).

6.0 INTERFERENCES

6.1 Some gamma isotopes emit gamma lines that may overlap with other isotopes. If the energies of the two isotopes are within the energy tolerance setting, the peaks may not be resolvable and may give a positive bias to the result. This problem is minimized by careful review of the peak search.

6.2 Soil samples may vary in density from the standard used for calibration. A density correction is applied to the "CAN" geometry. This correction was determined using solids with weights varying between 54 g and 192 g.

7.0 SAFETY PRECAUTIONS AND WARNINGS

7.1 Keep hands free from moving parts of canning device and gamma shields.

7.2 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

7.3 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

7.4 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.

7.5 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 100 cc aluminum cans with lids for soil and miscellaneous samples

8.1.2 10 cc Gelman Sciences Petri dish for soil, filters and miscellaneous samples

8.1.3 2 L and 500 mL Marinelli beakers for water samples

8.1.4 Air displacement pipettes

8.1.5 Can sealing tool

8.1.6 Graduated cylinder

8.1.7 25 cc VWR Petri for soil and miscellaneous samples

8.1.8 250 mL plastic jar for filters, soil, and miscellaneous samples

8.1.9 Hot plate

8.1.10 Teflon beakers and lids

8.1.11 1 L Marinelli beaker for soil samples

8.2 Instrumentation

8.2.1 High purity germanium detector, with associated electronics and data reduction software

8.2.2 NaI Detector with associated electronics and data reduction software

8.2.3 Top loader balance

9.0 REAGENTS, CHEMICALS, AND STANDARDS

9.1 NIST traceable mixed gamma standard in 100 cc aluminum can

9.2 NIST traceable mixed gamma standard in 2.0 L Marinelli beaker

9.3 NIST traceable mixed gamma standard in 0.5 L Marinelli beaker

9.4 NIST traceable mixed gamma standard in Gelman Sciences 10 cc Petri dish

9.5 NIST traceable mixed gamma standard in 13, 47 mm glass fiber filter composites in Gelman Sciences Petri dish.

9.6 NIST traceable mixed gamma standard in 0.4 L jar

9.7 NIST traceable mixed gamma standard in 0.25 L jar

9.8 NIST traceable mixed gamma standard in 1, 47 mm glass fiber filter

9.9 NIST traceable mixed gamma standard in Impregnated Charcoal Sample Cartridge.

9.10 NIST traceable mixed gamma standard in VWR (53 mm x 15 mm) Petri dish (approximately 25 cc)

9.11 NIST traceable mixed gamma standard in aqueous solution

9.12 NIST traceable mixed gamma standard in 1.0 L Marinelli beaker

9.13 NIST traceable mixed gamma standard in 20 mL liquid scintillation vial

9.14 16 M Nitric acid, reagent grade (HNO_3)

9.15 49% Hydrofluoric acid (HF)

9.16 12 M Hydrochloric acid, reagent grade (HCl)

9.17 5% Boric acid: Dissolve 50 g of H_3BO_3 per liter of DI water.

9.18 Nitric acid (8 M HNO_3): Prepare by cautiously adding a measured volume of concentrated nitric acid to an equal volume of DI water.

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 For soil samples, 500 g of sample should be collected, preferably in a plastic container to avoid breakage.

10.2 For water samples, 2 L of sample should be collected in a plastic container and preserved to a pH < 2 with nitric acid.

10.2.1 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should contact the Group Leader or Team Leader.

NOTE: If the analysis is requesting I-131 (or any other iodine isotopes) Analysis without preserving is acceptable. If a sample is preserved with acid without stabilizing the iodine, Iodine may volatilize and escape the solution as a gas.

10.2.2 If approved by the client, the analyst should adjust the pH with nitric acid to a pH < 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This acidification should be documented on a batch history sheet and attached to the batch paperwork.

10.3 For filters no preservation is necessary.

11.0 SAMPLE PREPARATION

11.1 Solid Sample Preparation.

11.1.1 Prepare the sample for gamma counting in accordance with SOP GL-RAD-A-021, Soil Sample Preparation for the Determination of Radionuclides.

11.1.2 Fill the appropriate container with sample prepared from step 11.1.1 using the following steps as a guideline:

11.1.2.1 If Ra-226 analysis is required, the sample is placed in a 100 cc can for in-growth.

NOTE: It is recommended that in-growth be allowed 20 days to quantify Ra-226. Shorter ingrowth periods can be used at the request of the client. However, shorter in-growth periods may decrease the accuracy of the data. If there is insufficient mass of sample to fill the 100 cc can, contact the Team or Group Leader.

11.1.2.2 If sufficient mass is available, homogenized samples should be placed in the 100 cc can. Determine the net weight of the sample. If the net weight is less than 54 g or greater than 192 g, contact the Team or Group Leader to determine the appropriate counting container. Record sample weight and date in AlphaLIMS and on sample container.

11.1.2.3 If there is insufficient sample to fill the 100 cc can, place sample in the 10 cc or 25 cc Petri dish, cap and seal. Record sample weight and date in AlphaLIMS and on sample container.

11.1.2.4 If there is insufficient sample to fill the 10 cc Petri dish, perform the following digestion process:

11.1.2.4.1 Weigh out an appropriate aliquot into a labeled Teflon beaker. Record this weight on the Queue sheet.

11.1.2.4.2 Add 10 mL of concentrated nitric acid to each sample.

11.1.2.4.3 Place samples on medium heat (approximately 300 qF) and cover each sample with a Teflon lid. Reflux all samples for 30 minutes.

11.1.2.4.4 Remove Teflon lids and add 5 mL concentrated hydrochloric acid and 10 mL hydrofluoric acid to each sample. Cover samples and reflux for 120 minutes.

11.1.2.4.5 Remove Teflon lids and allow samples to evaporate to dryness.

11.1.2.4.6 Add 5 mL of concentrated nitric acid and evaporate to dryness.

11.1.2.4.7 Repeat Step 11.1.2.4.6.

11.1.2.4.8 Add 5 mL of concentrated nitric acid to the dry samples. Add 1 mL of 5% boric acid. Place the samples back on the hot plate long enough so that the dried sample dissolves into solution.

11.1.2.4.9 Transfer solution to a 250 mL gamma container and dilute to 200 mL. Record the original sample mass and diluted volume on sample container.

Record the original sample mass on batch Queue sheet.

11.2 Water Sample Preparation

11.2.1 Place the appropriate labeled Marinelli beaker (typically 500 mL or 2 L) on a balance and tare the balance.

11.2.2 If less than approximately 1.1 L is available, sample should be poured into a 500 mL Marinelli beaker.

11.2.3 Transfer the appropriate volume to the tared container and record the volume of the sample on the Queue sheet.

NOTE: If there is insufficient sample to fill the Marinelli, record the exact amount of sample volume on the container and on the Queue sheet. Dilute the sample to the appropriate volume to maintain the calibration geometry. Record the volume the sample was diluted to on the sample container, also.

11.2.4 The MB should be recorded on the Queue sheet to be the same aliquot as the largest sample in the batch. An empty Marinelli beaker should be labeled as the MB and submitted with each batch of samples.

11.2.5 Submit the Marinellis and completed paperwork to the count room for gamma counting analysis.

11.3 Urine Sample Preparation

11.3.1 Refer to GL-RAD-B-030.

11.4 Preparation of Miscellaneous Matrices

11.4.1 Prepare the sample in accordance with GL-RAD-A-026 for The Preparation of Special Matrices for the Determination of Radionuclides.

11.4.2 If sample(s) was (were) received from the client in a container that matches a calibrated geometry, a direct count of the sample can be performed.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

Refer to GL-RAD-D-003.

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

Refer to GL-RAD-I-001.

14.0 ANALYSIS AND INSTRUMENT OPERATION

Refer to GL-RAD-I-001.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-010.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation and reduction take place in accordance with SOP GL-RAD-D-003 and GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Data are reviewed and packaged in accordance with GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as Quality Documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

20.1 USEPA. Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Method 901.1, August 1980.

20.2 Canberra Nuclear Genie System Spectroscopy, Applications and Display User's Guide, Vol. I and II, May 1991.

20.3 DOE EML Procedures Manual, HASL-300, 27 th Edition.

20.4 DOE EML Procedures Manual, HASL-300, 28 th Edition.

20.5 Manual for the Certification of Laboratories Analyzing Drinking Water. Criteria and Procedures Quality Assurance. Fifth Edition EPA 815-R-05-004 January 2005.

21.0 HISTORY

Revision 25: Type II to type I water.

Revision 24: Changed recovery limit for laboratory fortified blank from 90-100% to 90-110% in section 3.3.

Revision 23:Procedure updated to include requirements for drinking water samples.

Revision 22: Updated ingrowth period for Ra-226 to 20 days.

Revision 21: SOP revised to add Ra-226, NIST traceable gamma standards, and other clarifications.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF

RADIOMETRIC POLONIUM

(GL-RAD-A-016 REVISION 15)

PROPRIETARY INFORMATION

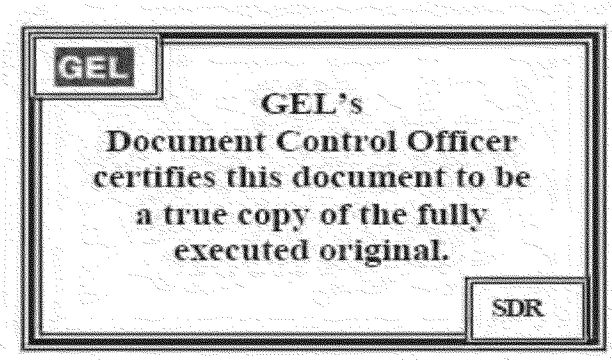

TABLE OF CONTENTS

1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF RADIOMETRIC POLONIUM....3
2.0 METHOD OBJECTIVE, PURPOSE AND SUMMARY....3
3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT....3
4.0 METHOD VARIATIONS....3
5.0 DEFINITIONS....3
6.0 INTERFERENCES....4
7.0 SAFETY PRECAUTIONS AND WARNINGS....4
8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION....4
9.0 REAGENTS AND STANDARDS....4
10.0 SAMPLE HANDLING AND PRESERVATION....5
11.0 SAMPLE PREPARATION....5
12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS....8
13.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE....8
14.0 PROCEDURE FOR ANALYSIS AND INSTRUMENTATION OPERATION....8
15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE....9
16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS....9
17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL....9
18.0 RECORDS MANAGEMENT....9
19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL....9
20.0 REFERENCES....9
21.0 HISTORY....9
APPENDIX 1....10

1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF RADIOMETRIC POLONIUM

2.0 METHOD OBJECTIVE, PURPOSE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic alpha emitting polonium in liquids, solids, and filters.

2.2 A solid sample is leached in concentrated nitric acid for a minimum of 2 hours. After centrifuging to remove the solids, the sample is diluted with DI water and treated as a liquid sample. Transuranic elements are scavenged by coprecipitation with calcium phosphate. This precipitate is dissolved and polonium is plated onto a nickel disk. This nickel disk is then mounted onto a metal disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for polonium is 1 pCi/L or 1 pCi/g.

3.2 Analysts training records are maintained as quality records. Refer to GL-QS-E-008 for Quality Records Management and Disposition.

4.0 METHOD VARIATIONS

Some variation may be necessary due to special matrices encountered in the laboratory. These variations may be used with approval from a Group or Team Leader. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

5.1 AlphaLIMS: GEL's Laboratory Information Management System.

5.2 Batch: Environmental samples prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.

5.3 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.

5.4 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

5.5 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.

5.6 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

5.7 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

5.8 Deionized (DI) Water: Type I water (Refer to GL-LB-E-016).

6.0 INTERFERENCES

Not applicable.

7.0 SAFETY PRECAUTIONS AND WARNINGS

7.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

7.2 Personnel handling Radioactive Materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.

7.4 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult senior qualified personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

8.1 Apparatus and Equipment

8.1.1 Polypropylene centrifuge tubes (50 mL)

8.1.2 Beakers (Glass and Teflon of various sizes)

8.1.3 Black electrical tape, 1" in width

8.1.4 Nickel plated disk, 7/8" diameter

8.1.5 Magnetic stir bars, 1" in length

8.1.6 Magnetic stir plates

8.1.7 Stainless steel tweezers

8.1.8 Metal disk, 29 mm diameter

8.1.9 Sample drying apparatus

8.1.10 Sample homogenizing apparatus

8.1.11 Watch glasses (various sizes)

8.1.12 Small plastic cups

8.1.13 Petri dishes

9.0 REAGENTS AND STANDARDS

9.1 Reagents

9.1.1 Hydrochloric acid, concentrated (12 M HCl)

9.1.2 Nitric acid, concentrated (16 M HNO_3)

9.1.3 Ammonium hydroxide, concentrated (14 N NH_4OH)

9.1.4 Ascorbic acid powder

9.1.5 Phosphoric acid, concentrated (H_3PO_4)

9.1.6 Hydrogen peroxide (30% H_2O_2)

9.1.7 1.25 M Calcium nitrate carrier: Dissolve 205 g of anhydrous calcium nitrate or 295 g hydrated calcium nitrate, $Ca(NO_3)_2 \cdot 4H_2O$, in 500 mL of DI water. Dilute to 1L with DI water.

9.1.8 1 M Hydrochloric acid: Add 84 mL of concentrated hydroch loric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.

9.1.9 Ethyl alcohol (80%): Dilute 800 mL of ethanol to 1 L with DI water.

9.1.10 6 M Hydrochloric Acid: Add 500 mL of concentrated hydrochloric acid to 500 mL of DI water.

9.2 Standards

9.2.1 NIST traceable standards: Po-209 and Po-210

9.2.2 Refer to GL-RAD-M-001.

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 Water samples should be collected in plastic bottles and preserved with concentrated nitric acid to pH < 2.

10.2 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should contact the Group Leader or Team Leader. If approved by the client, the analyst should adjust the pH with nitric acid to a pH < 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This acidification should be documented on a batch history sheet and attached to the batch paperwork.

10.3 If the sample has exceeded the hold time, the analyst should contact the Group Leader before continuing with the batch.

10.4 Solid samples require no preservation and may be shipped in any suitable container.

11.0 SAMPLE PREPARATION

NOTE: Aliquots may be estimated by using the count time estimator spreadsheet.

11.1 Aqueous Sample Preparation

11.1.1 Add an appropriate aliquot of sample to a labeled beaker. Prepare a Blank and LCS using DI water and a small amount of concentrated nitric acid to a pH < 2. The volume of DI water used should be the same as the largest volume of sample in the batch. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced on the Queue sheet. Record all aliquots on the Queue sheets.

11.1.2 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as

applicable. Reference batch Queue sheet and pull sheet for client requirements to determine appropriate tracer and spike.

11.1.2.1 For the determination of isotopic polonium, Po-209 is typically used as the tracer and Po-210 is typically used as the spike.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader, or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing on the Queue sheet.

11.1.3 Add 2 mL of 1.25 M calcium carrier, 1 mL of phosphoric acid, and 20 mL of 30% hydrogen peroxide to each sample. Stir thoroughly. Reflux on low setting for approximately 90 minutes.

11.1.4 Add concentrated ammonium hydroxide in a drop wise manner until visible white precipitate forms and the pH is between 8 and 9. Check the pH using a pH strip while precipitating.

11.1.5 Cover and reflux samples on low setting for approximately 90 minutes. Allow the sample to stand until precipitate settles.

11.1.6 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate.

11.1.7 Rinse the pellet with approximately 25 mL of DI water. Centrifuge. Decant and discard the supernate.

11.1.8 Rinse the beaker with approximately 25 mL 1 M hydrochloric acid, and cover and heat on a hot plate for several minutes to dissolve any residual calcium phosphate. Cool slightly and transfer rinse to the centrifuge tube. Cap and shake to dissolve the precipitate. Add 2 g of ascorbic acid powder to each sample. Cap and shake each centrifuge tube until the ascorbic acid dissolves. The sample should appear clear or slightly opaque.

NOTE: If any samples appear to contain iron, additional ascorbic acid should be added in 0.5 g increments. Cap and shake the centrifuge tube well after each addition. Continue until the sample has a clear or slightly opaque hue.

11.1.9 Transfer each sample to a clean, labeled Teflon beaker or new plastic cup. Rinse the centrifuge tube with approximately 25 mL of 1 M hydrochloric acid twice. Transfer each rinse to the Teflon beaker or plastic cup. (Final volume should be approximately 75 mL.)

11.1.10 Proceed to step 11.3.

11.2 Solid or Filter Preparation

11.2.1 If not already done, dry and homogenize the solid samples by performing GL-RAD-A-021. This step is not performed when analyzing filter media.

11.2.2 Measure an appropriate aliquot of solid (usually 0.2 g to 1.0 g) in a glass container. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced on the Queue sheet. Record all aliquots on the Queue sheet. The Blank and LCS aliquots should be recorded on the Queue sheet to be the same aliquot as the largest sample in the batch.

NOTE: Ashing of soils in a muffle furnace will result in significant polonium loss prior to analysis.

11.2.3 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference batch Queue sheet and pull sheet for client requirements to determine appropriate tracer and spike.

11.2.3.1 For the determination of isotopic polonium, Po-209 is typically used as the tracer, and Po-210 is typically used as the spike.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader, or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing on the Queue sheet.

11.2.4 Leach the samples in approximately 20 mL of concentrated nitric acid. More acid may be required for larger aliquots. Cover the samples and reflux on a low to medium setting for approximately 2 hours. Agitate the sample periodically to enhance the leaching process. Larger aliquots may need to reflux for a longer period of time. Do not allow the samples to go dry; add more acid if necessary.

11.2.5 Allow the samples to cool. Transfer each solid sample to a labeled centrifuge tube. Make sure to rinse the entire sample into the centrifuge tube using DI water. Centrifuge to separate the solid and leached portions of the sample. Decant the leachate back into a clean beaker. Rinse the solid phase with DI water. Centrifuge the sample and decant rinse into the beaker.

11.2.6 Dilute the leachate with DI water to a total volume of approximately 150 mL.

11.2.7 Proceed to step 11.1.3.

11.3 Nickel Disk Preparation

11.3.1 Prepare the nickel disk for use by taping one side of each disk with black electrical tape. Degrease the disk by first suspending each disk, one at a time, in concentrated nitric acid for approximately 5 seconds, followed by immediately suspending the disk in concentrated hydrochloric acid for approximately 5 seconds. Rinse each disk by suspending it in DI water. Blot the disk dry with a clean paper towel. Do not allow the disk to sit exposed to air for more than one hour.

NOTE: Excessive degreasing may result in low yields due to removal of the nickel plating.

11.3.2 Tape a magnetic stir bar to the back of each nickel disk with black electrical tape. Place a disk in each Teflon beaker or new plastic cup from step 11.1.9. Make sure the nickel side of the disk is facing up. Using a stir plate, stir the samples for at least 4 hours and no more than 16 hours at a low speed. DO NOT USE HEAT. Periodically check the samples to see if the disks are nickel side up and submerged in sample.

11.3.3 After plating is complete, remove each disk, one at a time, and thoroughly rinse with 6 M hydrochloric acid. Rinse the disc with DI water. Dry the disk by carefully blotting with a clean paper towel. Center each disk onto a labeled 29 mm flat planchet and place in a labeled Petri dish. Submit the samples for alpha spectroscopy counting.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

NOTE: Client contractual QC requirements override the requirements in this section.

12.1 Analyst and Method Verification Requirements

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Requirements

12.2.1 A method blank should accompany each batch of 20 or less samples. The reported value of the blank should be less than or equal to the CRDL (contract required detection limit).

12.2.2 The tracer added to all samples is used to calculate the method recovery. The method recovery of all samples should be between 15-125% when compared to the reference standard.

12.2.3 A duplicate sample should be run with each batch of 20 or less samples. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and QC DUP results are greater than 5 times the LLD or 100% if either result is less than 5 times the LLD.

12.2.4 A laboratory control sample (LCS) should be run with each batch of 20 or less samples. The recovery of the LCS should fall between 75-125%.

12.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the QC criteria from 12.2.1 through 12.2.4 cannot be satisfied, the analyst should inform the Group Leader and initiate a Data Exception Report as outlined in GL-QS-E-004.

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

For direction on calibration and instrument performance refer to GL-RAD-I-009.

14.0 PROCEDURE FOR ANALYSIS AND INSTRUMENTATION OPERATION

For analysis and instrument operation refer to GL-RAD-I-009.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

For maintenance of system refer to GL-RAD-I-010.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation, and reduction take place in accordance with GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Data are reviewed and packaged in accordance with GL-RAD-D-003 for Data Review, Validation and Data Package Assembly.

18.0 RECORDS MANAGEMENT

Data generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Laboratory waste is disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

20.1 Pb-210 Dating of Sediment Samples via Po-210 Alpha Spectrometry, Dr. Bill Burnett: Florida State University, 1992.

20.2 HASL-300 28 th Edition. Polonium in Water and Urine. PO-01-RC.

21.0 HISTORY

Revision 15: Made technical changes to section 5.8, 9.1.7, 11.3.2, and Appendix 1. Removed MS requirement from section 12.2. Updated section 16.0.

Revision 14: Removed acetone rinse. Added 6 M HCl rinse to step 11.3.3. Removed acetone from 9.1 Reagents and Chemicals. Added 6 M HCl to section 9.1 Reagents and Chemicals. Adjusted time allotted for spinning polonium samples.

Revision 13: Updated SOP to replace stainless steel disk with metal disk.

Revision 12: Made technical changes to sections 2.1, 2.2, 10.4, 11.2, 11.2.1, and 11.2.2. Replaced "Nonconformance Report" to "Data Exception Report" in section 12.3.

APPENDIX 1
Polonium

_____ Add 2 mL 1.25 M calcium carrier, 1 mL phosphoric acid, and 20 mL 30% H_2O_2

_____ Cover and reflux 90 minutes

_____ Add concentrated NH_4OH dropwise until pH is between 8 and 9

_____ Cover and reflux 90 minutes (allow to settle)

_____ Decant supernate, collect precipitate in c-tube and centrifuge

_____ Rinse pellet with 25 mL of DI water and centrifuge

_____ Rinse beaker with approximately 25 mL 1 M HCl, cover and reflux

_____ Transfer to c-tube, Add 2 g ascorbic acid powder

_____ Transfer to plastic cup

_____ Rinse c-tube with 25 mL 1 M HCl

_____ Rinse c-tube with 25 mL 1 M HCl

_____ Prepare nickel disk with black electrical tape

_____ Suspend disk in concentrated HNO_3, concentrated HCl, and DI water for 5 seconds each.

_____ Tape stir bar to disk and place in plastic cup

_____ Spin minimum 4 hours, maximum 16 hours

_____ Rinse with 6 M HCl and DI water

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

SOIL SAMPLE PREPARATION FOR THE DETERMINATION OF RADIONUCLIDES

(GL-RAD-A-021 REVISION 20)

PROPRIETARY INFORMATION

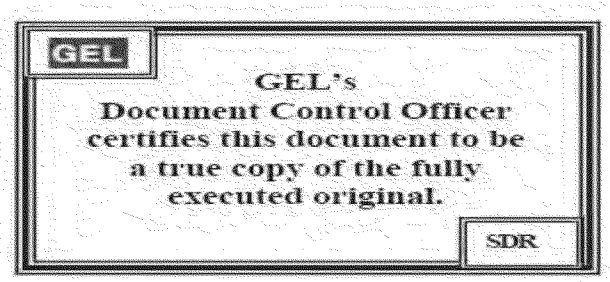

TABLE OF CONTENTS

1.0 STANDARD OPERATING PROCEDURE FOR THE SOIL SAMPLE PREPARATION FOR THE DETERMINATION OF RADIONUCLIDES 3
2.0 METHOD OBJECTIVE AND APPLICABILITY 3
3.0 SUMMARY 3
4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS 3
5.0 APPARATUS AND MATERIALS 3
6.0 SAMPLE COLLECTION AND PRESERVATION 4
7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE 4
8.0 SAMPLE PREPARATION PROCEDURES 4
9.0 CALCULATIONS AND DATA REDUCTION METHODS 7
10.0 QUALITY CONTROL REQUIREMENTS 7
11.0 CALIBRATION 7
12.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL 7
13.0 LABORATORY WASTE HANDLING AND DISPOSAL 8
14.0 REFERENCES 8
15.0 HISTORY 8
APPENDIX I: HGEO GROSS SAMPLE PRE-TREATMENT 9

1.0 STANDARD OPERATING PROCEDURE FOR THE SOIL SAMPLE PREPARATION FOR THE DETERMINATION OF RADIONUCLIDES

2.0 METHOD OBJECTIVE AND APPLICABILITY

This standard operating procedure provides the necessary instructions to conduct the preparation of soil samples for radionuclide determination.

3.0 SUMMARY

This procedure involves drying the soil at a temperature between 103 and 105 ºC. If that temperature would volatilize any components for which an analysis has yet to be run, a separate aliquot must be set aside for such analyses.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health, and Chemical Hygiene Plan, GL-LB-N-001.

4.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

4.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.

4.4 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

5.0 APPARATUS AND MATERIALS

5.1 Apparatus and Equipment

5.1.1 Metal cans, approximately quart and pint size

5.1.2 Steel balls, approximately 1" and 3/4" diameter

5.1.3 Sieve screens, 28 mesh

5.1.4 Paper funnels

5.1.5 100 cc aluminum cans

5.1.6 Aluminum loaf pans

5.1.7 SPEX steel grinding containers (various sizes)

5.1.8 Assorted tools and labware

5.2 Reagents, Chemicals, and Standards

5.2.1 Sand, clean

5.2.2 Deionized water (DI water)

5.3 Instrumentation

5.3.1 Paint can shaker, heavy duty

5.3.2 Analytical balance

5.3.3 SPEX Model 8515-115 Shatterbox

5.3.4 Drying oven

5.3.5 Hydraulic press

5.3.6 Retsch, Model BB-51, Jaw Crusher

5.3.7 Automatic or manual can sealer

6.0 SAMPLE COLLECTION AND PRESERVATION

A representative sample must be collected from a source of soil and should be large enough (50 to 100 g) so that adequate aliquots can be taken to obtain the required sensitivity. The container of choice should be plastic over glass to prevent loss due to breakage during handling. No preservation is required for solid samples.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to the technical manual provided with the paint shaker for information regarding equipment maintenance.

7.2 Refer to Instruction Manual for SPEX Shatterbox and Retsch Jaw Crusher operating instructions.

7.3 The analytical balance should be cleaned after use.

7.4 Refer to operating instructions for automatic or manual can sealer for information regarding equipment maintenance.

7.4.1 After receipt of new can sealer or when maintenance is performed on canner that could affect the proper sealing of the 100 cc gamma cans, perform a leak test on a can as follows:

7.4.1.1 Fill gamma can with water and seal.

7.4.1.2 Apply pressure on top and bottom of can and inspect for any visible signs of leakage.

8.0 SAMPLE PREPARATION PROCEDURES

8.1 Label a clean metal container with the laboratory sample number.

8.2 Weigh container. Record weight into the computerized soil prep balance log.

8.3 Transfer a representative aliquot from the sample to the labeled container.

8.3.1 When the amount of sample to be dried is not the entire contents of the sample container, refer to section 6.6 of SOP GL-LB-E-029 to ensure a representative sample aliquot is taken. This also ensures that the sample remaining is representative of the whole sample.

8.3.2 If the sample contains extraneous materials (i.e. rocks, twigs, vegetation) this shall be documented in the batch case narrative.

NOTE: Sample is dried at 103 to 105 qC. If this temperature will volatilize any component for an analysis that has yet to be run, a separate aliquot must be set aside for such analysis.

8.4 Enter the pre-oven sample weight into the computerized soil prep balance log. This weight represents the wet sample weight and container weight.

8.5 Place the container in a drying oven at a temperature between 103 and 105 qC for a minimum of four hours. (Normally overnight.)

NOTE: The time required to obtain a dry sample will vary depending on the type of material, size of sample, oven type and capacity, and other factors. The influence of these factors generally can be established by good judgment, experience with the materials being tested, and the apparatus being used.

8.6 Using heat resistant gloves, remove the sample from the oven and allow to cool.

8.7 Record weight into the computerized soil prep balance log. Replace sample in drying oven for a minimum of one hour.

8.8 Repeat step 8.7 until a constant weight is obtained. A constant weight is defined as a weight difference of less than about 0.1% (i.e. for a 20 g aliquot, the change in weight shuld be less than 0.02 g).

8.9 Homogenize the sample. This is normally accomplished by placing a lid on the container and placing the container in the industrial paint shaker. Depending on the matrix of the soil, it may be necessary to add several stainless steel balls inside the container to assist in homogenizing the sample. The length of time the sample remains on the shaker is dependent on the matrix of the sample, and normally ranges from 5 to 10 minutes. For solid samples that are composed of large particles, it may be necessary to reduce the particle size before homogenizing. This can be accomplished by placing the larger particles in the hydraulic press and applying enough pressure to break the particles into smaller pieces.

8.10 Remove the metal container from the shaker and allow to settle for several minutes.

8.11 Place the container in the sample preparation hood and remove the lid. If stainless steel balls were added to the container, they should now be removed. The stainless steel balls are discarded.

8.12 Determine an appropriate aliquot based on the analysis required. Normally, depending upon the required analysis, the sample will be passed through a 28 mesh sieve screen. For clients that require a smaller particle size, continue homogenizing samples per section 8.15.

8.13 Discard the unused portion of sample into the appropriate waste container (i.e. rocks or organic material). The soil sample is now ready for radiochemical analysis.

8.14 Place sample in appropriate containers (i.e. plastic bottle or vial, gamma can).

NOTE: If preparing a 100 cc gamma can, it is important that the can is properly sealed to ensure Ra-226 is quantified correctly. Ra-226 in soil samples is quantified by one of its daughter products (Bi-214). Ra-226 decays to Bi-214 through Rn-222, which is a gas, and must be isolated inside the can in order for equilibrium to be re-established.

8.14.1 For proper sealing of gamma can:

8.14.1.1 Place lid on 100 cc can and place can on base plate of the automatic or manual can sealer.

8.14.1.2 Raise the can until it is clamped firmly between the base plate and chuck by turning the can lifter handle as far as possible to the right until the handle locks itself against the frame.

8.14.1.2.1 If using the automatic can sealer push the on button. The flywheel will turn until the can sealing is complete.

8.14.1.2.2 If using the manual can sealer, turn the flywheel 21 turns until the second operational roll returns to its normal position, away from the chuck.

8.14.1.3 Lower the can by turning the can lifter handle to the left. Remove the sealed can from the base plate.

8.14.1.4 Visually inspect can after sealing for any defects. If defects are noticed that may affect the proper sealing of the can, remove contents from can and start over with a new can.

8.15 **Shatter Box (200 mesh sample)**

8.15.1 Take a portion of remaining sample (more than enough to complete the requested analysis) and further homogenize sample to approximately 200 mesh. This is accomplished by pulverizing sample in the shatterbox.

NOTE: The approximately 200 mesh is determined based on particle size study in shatterbox instruction manual.

8.15.2 Pulverize sample for a minimum of 5 minutes.

NOTE: Refer to Shatterbox Instruction Manual for operating procedure.

8.15.3 To prevent cross-contamination the dish and puck must be decontaminated prior to pulverizing the next sample.

8.15.3.1 Decontaminate the dish and puck by filling with approximately50 g of sand. Replace the lid.

8.15.3.2 Place the shatterbox in operation for approximately 1 to 2 minutes. Empty the sand from the container.

8.15.3.3 Dampen a clean paper towel with DI water and wipe the dish, puck and lid to ensure that all traces of sand are removed.

8.15.3.4 To check for contamination, perform a smear survey of dish and puck after each dish and puck decontamination with sand, and submit to Radiation Safety for counting.

8.15.3.5 A decontamination blank is analyzed monthly for gross alpha, beta, and gamma activity. The results of these analyses will be compared to historical data. When a decontamination blank is analyzed, it is pulverized post cleaning.

NOTE: To prevent corrosion, keep the entire container assembly dry.

8.15.4 Record all samples and decontamination blanks in the shatterbox logbook.

8.16 **Pulverizer**

8.16.1 Use the sample jaw crusher to prepare medium to extremely hard substances having a maximum input grain size of 35 mm.

NOTE: Refer to Retsch Jaw Crusher Type BB-51 Operating Instructions manual for operating procedure.

8.16.2 To prevent cross-contamination, the jaw crusher must be decontaminated prior to processing the next sample. Decontaminate the jaw crusher by processing approximately 200 g of pre-dried lava rock having a maximum input grain size of 35 mm. A decontamination blank is analyzed monthly for gross alpha, beta, and gamma activity. The results of these analyses will be compared to historical data.

9.0 CALCULATIONS AND DATA REDUCTION METHODS

The electronic balance program provides documentation of all necessary raw data.

Weights in AlphaLIMS are recorded to three places past the decimal point (Ex: 6.738, 314.197…).

9.1 If client requests results to be reported "as received" (based on wet weight), analysts will correct the "dry" aliquots back to wet weights using the "weight/loss aliquot correction report" link in AlphaLIMS. This report will be included, if necessary, in each analytical batch's raw data.

10.0 QUALITY CONTROL REQUIREMENTS

10.1 Method Specific Quality Control Requirements

10.1.1 When possible (i.e., there is sufficient sample available) for gamma analysis, a separate container will be prepared for counting to meet the duplicate sample requirement.

10.1.2 Refer to the specific isotope operating procedure for instructions concerning method quality control requirements.

10.2 Actions Required if the Quality Control Requirements Are Not Met

If any of the quality criteria cannot be satisfied, the analyst should inform the group leader and initiate a Data Exception Report as outlined in GL-QS-E-004 for Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items.

11.0 CALIBRATION

11.1 Balances are calibrated annually and verified daily in accordance with GL-LB-E-002.

11.2 Temperature monitoring devices are verified in accordance with GL-QS-E-007.

12.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

All data are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

13.0 LABORATORY WASTE HANDLING AND DISPOSAL

Laboratory waste is handled and disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

14.0 REFERENCES

14.1 ASTM C999-05, "Standard Practice for Soil Sample Preparation for Determination of Radionuclides," 1993 Annual Book of ASTM Standards, Vol 12.01, 2005.

14.2 Laboratory Sub-Sampling Procedure, GL-LB-E-029.

14.3 ASTM D6323-98 (2003) "Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities."

14.4 EPA's "Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples", EPA/600/r-03/027, November 2003.

15.0 HISTORY

Revision 20: Section 9.1 revised for clarification to comply with DOECAP audit finding.

Revision 19:Added client specific prep procedure as an appendix.

Revision 18: Changed 8.14-8.17 to 8.15-18.17 in section 8.12.

APPENDIX I: HGEO GROSS SAMPLE PRE-TREATMENT

This proposed procedure is for the preparation of SSFL soil samples for Hydrogeologic, Inc.

1.) Cover an area of bench top, within a HEPA filtered enclosure, with clean paper. Transfer the total raw sample to the paper and spread sample evenly across the surface. Samples may be contained in more than one sample container.

2.) Remove cultural/man-made materials from the sample if applicable, photograph, and place in a labeled container for storage. If no cultural/man/made materials are found, no photograph is required, provided that the laboratory documentation clearly notes that no such objects were found. Notify Project Manager if cultural/man-made materials are found in the samples.

3.) Label a clean metal container with the laboratory sample number. When large amounts of soil are being processed, samples may be dried in aluminum pans to improve effectiveness of complete drying. Note: Samples may be split into more than one drying vessel. Record weights separately until the sample is recombined.

4.) Weigh the containers and record weights into the soil prep balance log.

5.) Transfer the entire sample to the labeled container. When the amount of sample to be dried is not the entire contents of the sample container, document via a Data Exception Report. Any sample removed in this manner shall be done by taking a grab sample to minimize any loss of potentially volatile radionuclides.

6.) Enter the pre-oven sample weight into the soil prep balance log. This weight represents the wet sample weight and container weight.

7.) Place the container(s) in a drying oven at a temperature between 103 and 105 °C for a minimum of four hours. Record the time the sample was placed in the oven.

8.) Using protective heat resistant gloves remove the sample from the oven and allow cooling. Record the time the sample was removed from the oven.

9.) Record the weight in the soil prep balance log and place the sample back in the drying oven for a minimum of one hour. Record the time the sample was returned to the oven. Record the time the sample was removed from the oven for each interval.

10.) Repeat steps 8 and 9 until a constant weight is obtained. A constant weight is achieved when two subsequent weights agree within 1%.

11.) Break up the sample in preparation for passing through the sieves with the process. Transfer sample to an appropriate number of 1 gallon paint cans. Add 6- 1" stainless steel balls to each paint can to aid in the breakup of the samples. NOTE: Paint cans should not be filled above approximately 3/4 full. Overfilling will reduce the effectiveness of sample blending. Place the paint cans on the industrial paint can shaker for 5-10 minutes.

12.) Remove the container(s) from the paint can shaker and allow settling for several minutes.

13.) Place the container(s) in the sample in a ventilated preparation hood and remove the lid. If stainless steel balls were added to the container(s), they should now be removed and discarded.

14.) Pass entire dried sample through a 4-mesh sieve. Transfer material that will not pass through into a labeled container for storage. The analyst will evaluate the material to determine if they may be further broken up in order to be passed through the 4-mesh sieve.

15.) Pass the entire dried sample through a 28-mesh sieve. Any sample not passing through the 28-mesh sieve must be processed until it passes through the 28-mesh sieve. Contact the Group leader if these criteria cannot be met.

16.) Place sample back into the paint cans and mix on the paint can shaker for 5-10 minutes.

17.) From the <28-mesh sample, use the *'Cone and Quartering'* method described below to remove a representative sample fraction for gamma analysis (~2000 grams).

18.) ***Cone and Quartering'*** method - Sample is emptied out onto a non-contaminating smooth surface. Material is piled into a cone with a flattened top surface. Two top-to-bottom cuts are made through the cone at perpendicular angles to form four equal portions, or quarters. Two opposite quarters are compiled into a new cone, and the process is repeated until the proper sample mass is obtained. When sub-sampling using this method it is important to remove the entire quarters to be used. Process enough sample to prepare a new 1-Liter Marinelli beaker (~1600-1700 grams) and 100cc gamma can (~160-180 grams) geometries for gamma analysis.

19.) Record the weight of the gamma aliquots in the soil prep balance log. Some samples may need an equal portion of sample for archiving as noted by the project manager.

20.) Take a portion of remaining sample using the 'Cone and Quartering' method(more than enough to complete the requested analysis) and further homogenize sample to approximately 200 mesh. This is accomplished by pulverizing sample in the shatterbox (puck mill).[i]

Note: Some samples may be designated to prepare an equal portion of sample for archiving.

[i] The approximately 200 mesh is determined based on particle size study in shatterbox instruction manual. The number of replicates included in the study was determined based on guidance provided in chapter 6 of the MARLAP manual (Level B validation).

21.) Pulverize sample for 5 minutes. Refer to Shatterbox Instruction Manual for operating procedure.

22.) To prevent cross-contamination the dish and puck must be decontaminated prior to running the next sample. Decontaminate the dish and puck by filling with approximately 50 g of sand. Replace the lid and operate the shatterbox for 2 minutes. Empty the sand from the container.

23.) Dampen a clean paper towel with DI water and wipe the dish, puck and lid to ensure that all traces of the sand are removed. To check for gross contamination, perform a smear survey of dish and puck and submit to radiation safety office for counting.

24.) To monitor for low level contamination, process blanks will be analyzed. The process blanks will consist of an ICP/MS analysis for U-238 on DI water rinses of the grinding containers (500 mLs). Acceptable results of the blanks shall be less than MDL. Should a blank indicate the presence of U-238 at a level >MDL, it shall be documented and a review conducted to determine the impact on any data produced using the container in question.

Record all samples and decontamination blanks in the shatterbox logbook.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

GAMMA SPECTROSCOPY SYSTEM OPERATION

(GL-RAD-I-001 REVISION 19)

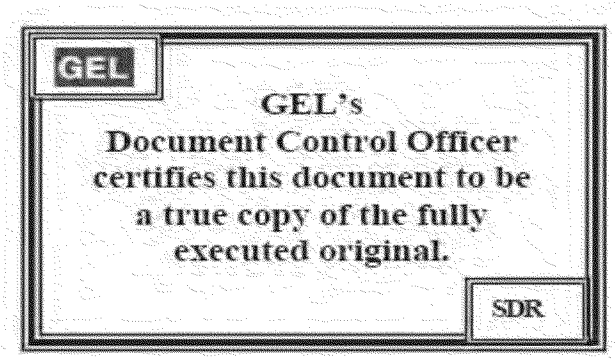

TABLE OF CONTENTS

1.0 STANDARD OPERATING PROCEDURE FOR GAMMA SPECTROSCOPY SYSTEM OPERATION....3
2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY....3
3.0 APPLICABLE MATRIX OR MATRICES....3
4.0 METHOD SCOPE, APPLICABILITY AND DETECTION LIMIT....3
5.0 METHOD VARIATIONS....3
6.0 DEFINITIONS....3
7.0 INTERFERENCES/LIMITATIONS....3
8.0 SAFETY PRECAUTIONS AND WARNINGS....3
9.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION....4
10.0 REAGENTS AND STANDARDS....4
11.0 SAMPLE HANDLING AND PRESERVATION....4
12.0 SAMPLE PREPARATION....4
13.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS....4
14.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE....4
15.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION....9
16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE....9
17.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS....10
18.0 POLLUTION/CONTAMINATION....10
19.0 DATA REVIEW, APPROVAL AND TRANSMITTAL....10
20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA....10
21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS....10
22.0 RECORDS MANAGEMENT....10
23.0 LABORATORY WASTE HANDLING AND DISPOSAL....10
24.0 REFERENCES....10
25.0 HISTORY....11

1.0 STANDARD OPERATINGPROCEDUREFORGAMMA SPECTROSCOPY SYSTEM OPERATION

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to cond uct the analysis for gamma isotopes using the Gamma Spectroscopy System.

2.2 Gamma emitting isotopes within the sample matrix are identified and quantified using gamma spectrometry. A sample aliquot is placed in a calibrated geometry and placed in the detector chamber. The germanium crystal therein produces a corresponding electrical pulse for the gamma photons that interact with the detector. The cumulative pulses are analyzed using software capable of quantifying gamma-emitting isotopes from the spectral data.

3.0 APPLICABLE MATRIX OR MATRICES

This is a nondestructive test for the measurement of gamma emitting isotopes in all matrices for which there is an available calibration standard.

4.0 METHOD SCOPE, APPLICABILITY AND DETECTION LIMIT

4.1 The aliquoted sample activity or sample position should be adjusted so that the detector system dead time remains less than 15%.

4.2 Method Detectable Activity: The MDA is based upon sample volume, instrument background, detector efficiency, count time and other statistical factors, as well as specific isotopic values such as abundance and half-life.

5.0 METHOD VARIATIONS

Not applicable

6.0 DEFINITIONS

6.1 Abundance: The combination of the isotopic decay branching ratio and the expected gamma emissions per disintegration of an isotope at a particular energy.

6.2 Key Line: The line chosen by the builder of the library to be the prominent line of the isotope. This line is used for the purposes of calculating activity, error and MDA.

6.3 AlphaLIMS: The Laboratory Information Management System used to store and report data.

6.4 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

7.0 INTERFERENCES/LIMITATIONS

7.1 Some gamma isotopes emit gamma lines that may overlap with those from other isotopes. If the energies of the two isotopes are within the energy tolerance setting, the peaks may not be resolvable and may give a positive bias to the result. This problem is minimized by careful review of the peak search.

8.0 SAFETY PRECAUTIONS AND WARNINGS

Follow safety precautions as outlined in GL-LB-N-001 for the Safety, Health and Chemical Hygiene Plan.

9.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

9.1 Apparatus and Equipment

9.1.1 Compaq/DEC Alpha Station with OpenVMS

9.1.2 Canberra Genie-ESP Application Software

9.1.3 High purity germanium detector

9.1.4 Pulse processing electronics

10.0 REAGENTS AND STANDARDS

10.1 Standards

10.1.1 NIST traceable mixed gamma standards in geometries and densities, closely approximating analytical samples, used to calibrate the instrument.

11.0 SAMPLE HANDLING AND PRESERVATION

Refer to GL-RAD-A-013 The Determination of Gamma Isotopes.

12.0 SAMPLE PREPARATION

Refer to GL-RAD-A-013 The Determination of Gamma Isotopes.

13.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

Refer to GL-RAD-A-013 The Determination of Gamma Isotopes.

14.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE

14.1 Calibration Standard

14.1.1 Mixed Gamma calibrations typically use a standard with 8-12 photons emitted over a range from approximately 45 keV to approximately 2000 keV.

14.1.2 Single nuclide calibrations typically use a standard comprised of the nuclide of interest.

14.2 Verification Standard

14.2.1 Mixed Gamma calibrations- A second source (from different manufacturer or if from the same manufacturer, a different lot number) is used for verification. The lines from Am-241, Cs-137 and Co-60 are used to verify the efficiency curve. These encompass the low, middle and high portions of the energy range.

14.2.2 Single nuclide calibrations – A second source (from a different manufacturer or if from the same manufacturer, a different lot number) is used for verification.

14.3 Standardization

14.3.1 High Voltage Adjust

14.3.1.1 See appropriate instrument manual for operation of electronics.

14.4 Calibration – Energy and efficiency calibrations are performed annually, upon initial instrument setup, after major repair or service, or when performance checks indicate a need.

NOTE: Expiration dates will match the last day of the month in which the calibration data was acquired.

14.4.1 Count Calibration Spectrum

14.4.1.1 Place the radioactive source on the detector.

14.4.1.2 Select **Calibration** ⇒ **Count a Calibration Standard** from the *Calibration* menu and click **OK**.

14.4.1.3 Enter the **Preset Live (secs):** in seconds and click **OK**. Count the standard until a minimum of 10,000 counts is acquired in each peak of interest.

14.4.2 Initial Energy & Shape Calibration

14.4.2.1 Select **Calibration** ⇒ **Initial Energy & Shape Calibration** from the *Calibration* menu and click **OK**.

14.4.2.2 Select the detector.

14.4.2.3 Select the **Certificate File** from the drop down list. Click **OK**.

14.4.2.4 From the *Energy Calibration* dialog box highlight one of the energy lines listed.

14.4.2.5 Move the cursor in the MCA window to the corresponding channel expected for that energy line.

14.4.2.5.1 The apex of the peak of interest should be at the expected channel.

14.4.2.5.2 From the *Energy Calibration* dialog box click the **Cursor** button.

14.4.2.6 Repeat the previous step until all energy lines listed have been referenced with a corresponding channel.

14.4.2.7 From the *Energy Calibration* dialog box select the **OK** button.

14.4.2.8 The system will ask "Do you want to do a full energy and shape calibration?" Select **YES**.

14.4.2.9 The energy and shape calibrations will now be performed with all of the lines from step 14.4.2.6. Verify the energy and shape curve generated. Select **OK** to continue or **Cancel** to abort the calibration.

14.4.2.10 A new page will appear with the Energy Calibration Report and the FWHM Calibration Report. Review the columns marked difference. For the energy calibration, the absolute value of the difference must be less than 1.0 and for the FWHM calibration, the absolute value of the difference must be less than 0.5. Regardless of the results, select **Dismiss.**

14.4.2.11 A new pop-up screen will appear. If the results from the previous step were less than a 0.2 keV difference, select **OK**. If the results were greater than a 0.2 keV difference select **Cancel** and begin the energy calibration process again at step 14.4.1.

14.4.3 Energy Re-Calibrate

14.4.3.1 Select **Calibrate Œ Re-Calibrate Œ Energy and Shape Calibration** from the main menu and click **OK**.

14.4.3.2 Select the detector.

14.4.3.3 Select the certificate file and select the **OK** button.

14.4.3.4 The energy and shape calibrations will now be performed with all of the lines from step 14.4.2.6. Verify the energy and shape curve generated. Select **OK** to continue or **Cancel** to abort the calibration.

14.4.3.5 A new page will appear with the Energy Calibration Report and the FWHM Calibration Report. Review the columns marked difference. For the energy calibration, the absolute value of the difference must be less than 1.0 and for the FWHM calibration, the absolute value of the difference must be less than 0.5. Regardless of the results, select **Dismiss.**

14.4.3.6 A new pop-up screen will appear. If the results from the previous step were less than a 0.2 keV difference, select OK. If the were greater than a 0.2 keV difference select Cancel and begin the energy calibration process again. If it fails after re-calibration contact Group or Team Leader for further instructions.

14.4.4 Efficiency Calibrate

14.4.4.1 Select **Calibrate Œ Efficiency Calibrate** from the main menu.

14.4.4.2 Select the geometry that represents the standardized radioactive source and click **OK**. If the geometry doesn't exist select **Create New Geometry**, enter the name of the new geometry and select **OK**.

14.4.4.3 Select the certificate for the calibration standard and select the **OK** button.

14.4.4.4 The efficiency calibration curve will be displayed for review. Select Empirical fit, and Log scale.

14.4.4.5 To accept the calibration select **OK**, or select **Cancel** to abort.

14.4.4.6 Dismiss the Calibration report displayed to complete the calibration procedure.

14.4.4.7 In the DECterm type **EFFPlot,** then press ENTER the type **EFFPRINT** then hit ENTER. This will print the efficiency curve.

14.4.5 Efficiency Verifications

14.4.5.1 Verification counts are performed as a normal sample count starting at step 15.2.3 of this SOP.

14.4.5.2 No batch ID is assigned to verification counts, typically "VER" is used.

14.4.5.3 Select the only sample identification available regardless of how it is named.

14.4.5.3.1 You may be asked if you would like to extend the count. Select **NO**.

14.4.5.3.2 When the screen to enter the sample information appears (step 15.2.8), use the date and time indicated on the manufacturer's certificate file for decay correction and change the sample identification using the following naming convention:
VER_DETECTOR_GEOMETRY, for example VER_GAM01_CAN.

14.4.5.4 Once the count has completed, in the DECterm, type "@print_virtual sample, where sample equals the same identification used in step 14.4.5.3.2. This will print out the raw data of the verification count.

14.4.5.5 Several pages will print out. The only pages needed are the background-subtracted peak report, which should be the first page, and the nuclide line activity report.

14.4.5.6 Place the results from the "Decay Corr" column into the appropriate Master Verification Spreadsheet located at S:\RAD\FORMS\EFF_VER where S:= sdrive on 'radserver' under the column named **Measured Activity**.

14.4.5.7 If necessary, enter the emission rate for the standard used for verification on the Master Verification Spreadsheet. This can be found on the manufacturer's certificate file for the standard. The spreadsheet will then calculate the **Calibrated Activity**. If a column for the emission rate does not exist on the spreadsheet, the **Calibrated Activity** can be calculated by using the values from the Decay Correct Source page in Alpha LIMS

(http://prodsvr01.gel.com:7778/pls/lims/de_ref_material.decay_correction).

14.4.5.8 The percent difference between the **Calibrated Activity** and **Measured Activity** is calculated by the spreadsheet and is displayed under the column marked **Difference**. The verification is considered acceptable if all values in the **Difference** column are less than 10%. If the **Difference** is 10% or greater, the verification is considered invalid and must be performed again. If two verifications fail notify Group Leader or Team Leader for further action.

14.5 Performance Checks

14.5.1 Daily Quality Control Calibration Check (QCC)

14.5.1.1 The QCC should be counted daily or prior to sample counting. If no samples are being counted this check is not required.

14.5.1.2 Load the QCC check source on the detector(s). If multiple QCC checks are being started skip to step 14.5.1.5.

14.5.1.3 From the PROcount window, select **QC Calibration Check.**

14.5.1.4 Select the detector and select **OK**.

14.5.1.5 To start multiple QCC checks at once, select **QC Multi Calibration Checks** from the PROcount window.

14.5.1.6 Highlight each detector you wish to start by clicking once on the detector name. Once you have highlighted all of the detectors you wish to start, select **OK**.

14.5.2 Daily Quality Control Background Check (QCB)

14.5.2.1 The QCB should be counted daily or prior to sample counting. If no samples are being counted this check is not required.

14.5.2.2 Ensure the detector shield(s) are empty prior to running the QCB. If multiple QCB checks are being started, skip to step 14.5.2.5.

14.5.2.3 From the PROcount window, selec **t QC Background Check.**

14.5.2.4 Select the detector and select OK.

14.5.2.5 To start multiple QCB checks at once, select **QC Multi Background Checks** from the PROcount window.

14.5.2.6 Highlight each detector you wish to start by clicking once on the detector name. Once you have highlighted all of the detectors you wish to start, select OK.

14.5.3 Weekly Environmental Background

14.5.3.1 Ensure the detector shield(s) is (are) empty. The same process will be used to start single and multiple weekly environmental background counts.

14.5.3.2 Select **Count Start MultipleBackgrounds** from the PROcount window.

14.5.3.3 highlight each detector you wish to start by clicking once on the detector name. Once you have highlighted all of the detectors you wish to start, select **OK.**

14.5.4 Generating the Daily and Weekly Check Reports

14.5.4.1 Daily check reports will generate every day following the completion of the QCC and QCB counts for each detector that will be in operation.

14.5.4.2 In the DECterm, type the command " **@QA_REPORT D**" then hit ENTER.

14.5.4.3 Weekly check reports will be completed once per week, typically Monday, following the completion of the weekly background subtraction counts.

14.5.4.4 In the DECterm, type the command " **@QA_REPORT B**" then hit ENTER.

15.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

15.1 Prepare the sample as outlined in GL-RAD-A -013 for The Determination of Gamma Isotopes.

15.2 Sample Counting

15.2.1 Prior to starting a sample count the detector used must be scanned into AlphaLIMS. In a web browser, enter the following address:

http://prodsvr01.gel.com:7778/pls/lims/inst_instrument.start_count

15.2.2 Each sample and detector are labeled with a Universal Product Code (UPC). First scan the UPC code for the detector and then scan the UPC code for the sample. Continue doing so for any additional sample counts. Once this has been done, select Submit on the web page.

15.2.3 Load the sample on the detector.

15.2.4 Select **Count Start a Count** from the *ProCount Main Menu.*

15.2.5 Select a detector and select **OK**.

15.2.6 Enter the batch to be started and select OK.

15.2.7 Select the sample to be counted.

15.2.8 Enter the sample specific information into the Sample Information screen and select OK.

15.2.9 Select the Analysis Sequence file used for analysis and select OK.

15.2.10 Select the counting geometry and select OK.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-010 for Counting Room Instrumentation Maintenance.

17.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

Data recording, calculation and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

18.0 POLLUTION/CONTAMINATION

Ensure all samples are bagged prior to counting to prevent instrument contamination.

19.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

Refer to GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data might require instrument maintenance, re-analysis, using a new spike mix, or a more complex set of actions. When trouble-shooting measures (refer to Section 21) fail to bring an analytical process or data into control, a data exception report and/or corrective action should be initiated in accordance with GL-QS-E-004.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting the instrument is a function of analyst experience. In-house service is obtained from GEL's Group Leader or other qualified personnel. If vendor assistance is needed, then the appropriate vendor is contacted. Maintenance logbooks are kept for each instrument and contain entries for both routine and non-routine maintenance procedures.

22.0 RECORDS MANAGEMENT

22.1 Each sample analysis that is performed is documented in the instrument run log in accordance with GL-LB-E-009 for Run Logs.

22.2 All raw data printouts, calculation spreadsheets, and batch checklists are filed with the sample data for archival in accordance with GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

22.3 Instrument maintenance is recorded in accordance with GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices.

22.4 Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

23.0 LABORATORY WASTE HANDLING AND DISPOSAL

Laboratory waste is disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

24.0 REFERENCES

24.1 United States Department of Energy, Environmental Measurements Laboratory, HASL-300 The Procedures Manual of the Environmental Measurements Laboratory, 28th Edition, "Gamma Radioassay," Ga-01-R (Vol. 1), February 1997.

24.2 United States Environmental Protection Agency, Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Method 901.1, August 1980.

24.3 American National Standards Institute, American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides, ANSI N42.14-1999.

24.4 Canberra Model 480720 ProCount-ESP Users Manual, September 2000.

24.5 Canberra Model 480726 Genie-ESP System Users Manual, September 2000.

24.6 Canberra Model 480198 Genie VMS Users Manual, 2000.

24.7 ASTM, International, Standard Practice for Setup, Calibration, and Quality Control of Instruments Used for Radioactive Measurements, D7282-6, Nov. 2010.

25.0 HISTORY

Revision 15: Procedural updates made to SOP to reflect the process currently being used.

Revision 16: Added criteria for acceptance of FWHM calibration.

Revision 17: Updated sections 14.4.3.6 and 14.4.5.2 for clarification.

Revision 18: Added note to section 14.4 to clarify instrument calibration expiration dates.

Revision 19: Revised to include new GL-RAD-D-006 for calculations.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

ALPHA SPECTROSCOPY SYSTEM

(GL-RAD-I-009 REVISION 14)

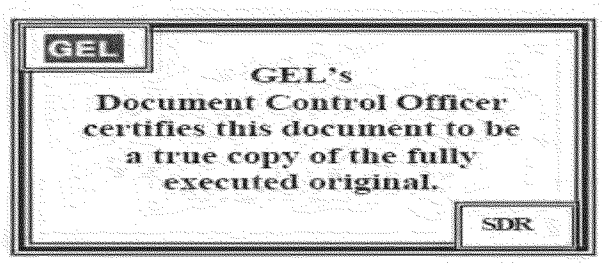

TABLE OF CONTENTS

1.0 STANDARD OPERATING PROCEDURE FOR ALPHA SPECTROSCOPY SYSTEM3
2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY 3
3.0 APPLICABLE MATRICES 3
4.0 METHOD SCOPE, APPLICABILITY AND DETECTION LIMIT.....3
5.0 METHOD VARIATIONS.....3
6.0 DEFINITIONS.....3
7.0 INTERFERENCES/LIMITATIONS.....4
8.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS.....4
9.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION.....4
10.0 REAGENTS AND STANDARDS.....5
11.0 SAMPLE HANDLING AND PRESERVATION.....5
12.0 SAMPLE PREPARATION.....5
13.0 QUALITY CONTROL SAMPLES.....5
14.0 STANDARDIZATION AND CALIBRATION.....5
15.0 OPERATING PROCEDURE.....12
16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE.....15
17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL.....16
18.0 POLLUTION/CONTAMINATION 16
19.0 DATA RECORDING, CALCULATIONS, AND REDUCTION METHODS.....16
20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA...16
21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS.....16
22.0 RECORDS MANAGEMENT.....16
23.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL.....16
24.0 REFERENCES 16
25.0 HISTORY.....17
APPENDIX 1: TROUBLESHOOTING.....18

1.0 STANDARD OPERATING PROCEDURE FOR ALPHA SPECTROSCOPY SYSTEM

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

This method establishes the procedures for general use and calibration of the Canberra Alpha Spectroscopy System used to obtain and analyze alpha spectra for samples containing single or multiple alpha-emitting radionuclides. The operation of the Canberra Alpha Analyst and model 7401 alpha spectrometers is discussed. This method also describes how specific radionuclides are identified and quantified from the spectral data.

This procedure also outlines the required scheduled maintenance and performance checks for the instruments. In order to assure the optimum performance of count room instrumentation, it is necessary to perform regularly scheduled maintenance and instrument checks. The instrument checks include energy and efficiency calibration, which are conducted once a month, backgrounds, which are conducted weekly, and daily pulser checks. The scheduled maintenance provides a means of maintaining instrument performance, while minimizing the "down time" due to instrument failure and repair.

3.0 APPLICABLE MATRICES

Applies to all matrices.

4.0 METHOD SCOPE, APPLICABILITY AND DETECTION LIMIT

The procedure is not specific to one particular method. For method scope, applicability or detection limit refer to the method specific analytical standard operating procedure.

5.0 METHOD VARIATIONS

Not applicable.

6.0 DEFINITIONS

6.1 Average Efficiency: The average of the calculated efficiency of each isotope contained on the efficiency standard.

6.2 Background: Those counts that can be observed and thereby, allowed for by measuring a blank background planchet. These counts are attributable to environmental radioactivity, recoil contamination of the detector, electronic noise pulses, etc.

6.3 Efficiency: A percent of decay events from a standard radioactive source that are seen and measured by a detector.

6.4 Energy Calibration Offset: The energy (keV) that corresponds to the first channel on the Multichannel Analyzer for each chamber.

6.5 FWHM (Full Width Half Maximum): The full width of an alpha peak distribution measured at half the maximum peak height.

6.6 Peak Area: The number of counts contained within an alpha peak.

6.7 Peak Energy: The energy (keV) measured at the center of the alpha peak.

6.8 Peak Resolution: The FWHM value of the alpha peak.

6.9 Performance Check: any operation performed on an instrument to verify its ability to conform to required specifications

6.10 PIPS detector: Passivated Implanted Planar Silicon

6.11 Scheduled Maintenance: any operation performed on an instrument to prevent premature equipment failure

6.12 Traceable Calibration Standard: A calibrated radioactive source, with stated accuracy, whose calibration is certified by or to NIST (National Institute of Standards and Technology) or an equivalent organization.

6.13 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

6.14 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.

7.0 INTERFERENCES/LIMITATIONS

For analyses requiring isotope specific analyses (i.e. U-238, Pu-238) chemical separations are performed during sample preparation to remove unwanted counting interferences.

8.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

8.1 Refer to the Radioactive Material Handling Procedure (GL-RAD-S-004) for instructions on the handling of radioactive samples.

8.2 Refer to the Laboratory Waste Management Plan (GL-LB-G-001) for instructions on proper disposal of materials.

8.3 The detector bias supply must remain off, until the detector chamber reaches the normal operating vacuum, to prevent damage to the surface barrier detectors.

8.4 Turning off, or loss of power to, the vacuum pumps could lead to oil contamination of the alpha detectors. Therefore, all detectors must be brought to atmospheric pressure prior to turning the vacuum system off or immediately after a loss of power.

8.5 Follow the manufacturer's instructions for set up, intercomponent connections, and preliminary testing of the equipment. Observe all of the manufacturer's limitations and precautions.

8.6 Never exceed the manufacturer's recommended operating voltage for the detector; this may lead to detector damage.

9.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

9.1 Canberra model 7401 Alpha Spectrometer

9.2 Canberra model 7200 Dual Alpha Analyst Spectrometer

9.3 Canberra model 7200 Controller

9.4 DEC/Compaq Alpha workstation or equivalent

9.5 Vacuum Pump

9.6 AMX analog multiplex or module

9.7 Acquisition Interface Module

9.8 ADC (analog to digital converter)

9.9 Vacuum pump and filtration rig

9.10 Disposable filter funnels (containing 25 µm filters with 0.1 mm pore size)

9.11 Stainless steel disks (29 mm diameter)

9.12 Stainless steel tweezers

10.0 REAGENTS AND STANDARDS

10.1 Traceable Calibration Standard - The actual standard is dependent upon the sample geometry being calibrated.

10.2 Vacuum Pump Oil

10.3 Silicone grease

11.0 SAMPLE HANDLING AND PRESERVATION

Not applicable.

12.0 SAMPLE PREPARATION

Not applicable.

13.0 QUALITY CONTROL SAMPLES

Not applicable.

14.0 STANDARDIZATION AND CALIBRATION

NOTE: Refer tp GL-RAD-M-001 for guidance on the preparation of calibration sources for alpha spectroscopy.

14.1 Energy and Efficiency Calibration (Monthly checks for Alpha analyst detectors)

14.1.1 Alpha calibration standards are counted once each calendar month to update the detector energy and efficiency calibrations.

14.1.2 From the AMS Procedure window, select Displays then Chamber Status; ensure detectors are free for use.

14.1.3 Using a pair of tweezers, carefully position the appropriate calibration standard into each counter, taking care to center the calibration standard beneath the detector face and ensure the sample shelf is in the proper location.

14.1.4 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.

14.1.5 Close the chamber doors.

14.1.6 From the AMS Procedure window, select **Count** then **Primes**.

14.1.7 Enter a list of the chambers that you are starting and click **OK** or press **Enter**.

14.1.8 The detectors will automatically evacuate air from the chamber and apply a detector bias. The system then starts data acquisition on all alpha counters and counts the standards for a pre-determined time suitable to achieve greater than 10,000 counts in each applicable region of interest (Gd-148, Np-237, and Cm-244) typically 2 to 4 hours.

14.1.9 When the count is complete, the detector bias will automatically be turned off and the chamber vented to atmosphere.

14.1.10 When the calibration count is completed, proceed with section 14.3.

14.2 Energy and Efficiency Calibration (Monthly checks for model 7401 detectors)

14.2.1 Alpha calibration standards are counted once each calendar month to update the detector energy and efficiency calibrations.

14.2.2 From the DECterm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting System Main Menu is displayed, proceed with 14.2.3. If the Sample Counting System Main Menu is not displayed, consult with the Group Leader or their designee.

14.2.3 Select **1) Sample Counting** to access the Sample Counting Menu.

14.2.4 From Sample Counting Menu, Select **2) List Status of Detectors**; ensure detectors are free for use. Press **Return** to exit.

14.2.5 Using a pair of tweezers, carefully position the appropriate calibration standard into each counter, taking care to center the calibration standard beneath the detector face and ensure the sample shelf is in the proper location.

14.2.6 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.

14.2.7 Close the chamber doors and start evacuation of the chambers in accordance with section 15.5.

14.2.8 After normal operation vacuum is achieved, turn on detector bias.

14.2.9 Select **1) Count a New Sample** to access the Alpha Counting Menu.

14.2.10 Select **3) Monthly Calibration Check**.

14.2.11 Verify that all detector bias supplies are on, all pulsers are off. Enter a list of detectors to start, and press **Return**. The system then starts data acquisition on all alpha counters and counts the standards for a pre-determined time suitable to achieve greater than 10,000 counts in each applicable region of interest (Gd-148, Np-237, and Cm-244) typically 2 to 4 hours.

14.2.12 When the calibration count is completed, proceed with section 14.3.

14.3 Manually Processing the Monthly Calibrations

14.3.1 Proceed to DECterm VMS prompt. If the sample Counting System menu is still displayed on the DECterm, then exit to the DECterm VMS prompt by pressing **R** and **Return**.

14.3.2 Check the contents of the file **NAMES.DAT** by typing **EDIT NAMES.DAT**. Edit the contents of the file so it contains the entries **W###-W###**, depending on the detectors to be processed. When the contents of the file are correct, press **Control Z** followed by **Quit** if the changes are not to be saved, or Exit if the changes are to be save. Type **RANGE # #** to indicate which banks to show on the supervisor's action report.

14.3.3 At the prompt, type **Process** to start the processing of the calibrations.

14.3.4 At the **Initial or Update calibration? (I/U)** prompt, enter **U** to update the calibration or enter **I** to perform an initial calibration. Note that an initial calibration is done only under specific circumstances, such as initial setup of a counter. Consult the Group Leader or designee prior to performing an initial calibration. If a calibration update was chosen, the operator will be asked to verify the update energy and efficiency parameters for each detector.

14.3.5 Initial Calibration Only

14.3.5.1 At the prompt use the mouse to position the cursor over the center of the specified nuclide on the spectrum display. Press **Return**.

14.3.5.2 Repeat for each nuclide.

14.3.6 Proceed with section 14.4.

14.4 Reviewing Monthly Calibration Report

14.4.1 After the monthly calibration data is processed, a supervisor's action report will be printed. Review the report for any out of control condition. Contact Group Leader or designee for out of control conditions. Detector should be removed from service if any of the following conditions exist: ABOVE/BELOW PSAREA, NLACTIVITY, ECOFFSET, ECSLOPE, AVRGEFF, FWHMCONST, PSENERGY, PSFWHM.

NOTE: Refer to GL-RAD-I-012, Managing Statistical Data in the Radiochemistry Laboratory, for guidance on locking out detectors.

14.4.2 Update detector status board.

14.5 Daily Pulser Checks for Alpha analyst detectors

14.5.1 Daily pulser checks performed daily, prior to counting samples, to verify the proper operation of the detectors. Peak centroid, Pulser count rate, and peak FWHM are monitored and stored in quality assurance files.

14.5.2 From the AMS Procedure window, select **Displays** then **Chamber Status**; ensure detectors are free for use.

14.5.3 From the AMS Procedure window, select **Count** then **Pulsers**.

14.5.4 Enter a list of the chambers that you are starting and click **OK** or press **Enter**.

14.5.5 The detectors will automatically evacuate air from the chamber and apply a detector bias. The system then starts data acquisition on all alpha counters and counts the pulsers for 5 minutes.

14.5.6 When the count is complete, the detector bias will automatically be turned off and the chamber vented to atmosphere.

14.5.7 When the daily pulser count is completed, the data may be automatically processed and a Supervisor's Action Report is generated. If a report is generated, proceed with section 14.8. If a report is not generated, proceed with section 14.7.

14.6 Daily Pulser Checks for model 7401 detectors

14.6.1 Daily pulser checks performed daily, prior to counting samples, to verify the proper operation of the detectors. Peak centroid, Pulser count rate, and peak FWHM are monitored and stored in quality assurance files.

14.6.2 From the DECterm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting System Main Menu is displayed, proceed with 14.6.3. If the Sample Counting System Main Menu is not displayed, consult the Group Leader or their designee.

14.6.3 Select **1) Sample Counting** to access the Sample Counting Menu.

14.6.4 From Sample Counting Menu, **Select 2) List Status of Detectors**; ensure detectors are free for use. Press **Return** to exit.

14.6.5 If needed start evacuation of the chambers in accordance with section 15.5.

14.6.6 After normal operating vacuum is achieved, turn on detector bias and activate the detector pulser.

14.6.7 Select **1) Count a New Sample** to access the Alpha Counting Menu.

14.6.8 Select **2) Daily Pulser Check**.

14.6.9 Verify that all pulser and bias supplies are enabled for each detector. Enter a list of detectors to start. The system then starts data acquisition on all alpha counters and counts the pulsers for 5 minutes.

14.6.10 When the daily pulser count is completed, the data may be automatically processed and a Supervisor's Action Report generated. If a report is generated, proceed with section 14.8. If a report is not generated, proceed with section 14.7.

14.7 Manually Processing the Daily Pulser Checks

14.7.1 Proceed to the DECterm VMS prompt. If the Sample Counting System menu is still displayed on the DECterm, then exit to the DECterm VMS prompt by pressing **R** and **Return**.

14.7.2 Check the contents of the file **NAMES.DAT** by typing **EDIT NAMES.DAT**. Edit the contents of the file so it contains the entries **D###-D###**, depending on the number of detectors to be processed. When the contents of the file are correct, press **Control Z** followed by **Quit** if the changes are not to be saved, or **Exit** if the changes are to be saved. Type **RANGE # #** to indicate which banks to show on the supervisor's action report.

14.7.3 At the $ prompt, type **Process** to start the processing of the Daily Pulser Checks. The program proceeds to automatically process the pulser data one counter at a time.

14.7.4 Proceed with section 14.8.

14.8 Reviewing Daily Pulser Report

14.8.1 After the data is processed, a Supervisor's Action Report and Missing QA Report will be printed. Review the Supervisor's Action Report for any out of control conditions. Contact Group Leader or designee immediately for out of control conditions. Detector should be removed from service for the day if any of the following conditions exist: ABOVE/BELOW PSFWHM, PSENERGY, PSCENTRD, PSCTSS. Review the Missing QA Report for any detectors that may not have run Daily Pulser Checks. Perform section 14.6 or 14.5 as needed on any detectors listed on the Missing QA Report.

NOTE: Refer to GL-RAD-I-012, Managing Statistical Data in the Radiochemistry Laboratory, for guidance on locking out detectors.

14.8.2 Update detector status board.

14.9 Weekly Background Checks for Alpha Analyst detectors

14.9.1 Blank planchets are counted once each week to update the detector background counts.

14.9.2 From the AMS Procedure window, select **Displays** then **Chamber Status**; ensure detectors are free for use.

14.9.3 Using a pair of tweezers, carefully position the appropriate background planchets into each counter, taking care to center the planchet beneath the detector face and ensure the sample shelf is in the proper location.

14.9.4 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.

14.9.5 Close the chamber doors.

14.9.6 From the AMS Procedure window, select **Count** then **Backgrounds**.

14.9.7 Enter a list of the chambers that you are starting and click **OK** or press **Enter**.

14.9.8 The detectors will automatically evacuate air from the chamber and apply a detector bias. The system then starts data acquisition on all alpha counters and counts the backgrounds for the predetermined count time.

14.9.9 When the count is complete, the detector bias will automatically be turned off and the chamber vented to atmosphere.

14.9.10 When the background is completed, the data may be automatically processed and a Supervisor's Action Report generated. If a report is generated, proceed with section 14.12. If a report is not generated, proceed with section 14.11.

14.10 Weekly Background Checks for model 7401 detectors

14.10.1 Blank planchets are counted once each week to update the detector background counts.

14.10.2 From the DECterm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting System Main Menu is displayed, proceed with 14.10.3. If the Sample Counting System Main Menu is not displayed, consult the Group Leader or their designee.

14.10.3 Select **1) Sample Counting** to access the Sample Counting Menu.

14.10.4 From Sample Counting Menu, Select **2) List Status of Detectors**; ensure detectors are free for use. Press Return to exit.

14.10.5 Using a pair of tweezers, carefully position the appropriate background planchets into each counter, taking care to center the planchet beneath the detector face and ensure the sample shelf is in the proper location.

14.10.6 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.

14.10.7 Close the chamber doors and start evacuation of the chambers in accordance with section 15.5.

14.10.8 After normal operating vacuum is achieved, turn on detector bias.

14.10.9 Select **1) Count a New Sample** to access the Alpha Counting Menu.

14.10.10 Select **3) Backgrounds**.

14.10.11 Verify that all detector bias supplies are on, all pulsers are off. Enter a list of detectors to start, and press **Return**. The system then starts data acquisition on all alpha counters and counts the backgrounds for the predetermined count time.

14.10.12 When the background count is completed, the data may be automatically processed and a Supervisor's Action Report generated. If a report is generated, proceed with section 14.12. If a report is not generated, proceed with section 14.11.

14.11 Manually Processing Weekly Backgrounds

14.11.1 Proceed to the DECterm VMS prompt. If the Sample Counting System menu is still displayed on the DECterm, then exit to the DECterm VMS prompt by pressing **R** and **Return**.

14.11.2 Check the contents of the file **NAMES.DAT** by typing **EDIT NAMES.DAT**. Edit the contents of the file so it contains the entries **B###-B###**, depending on the number of detectors to be processed. When the contents of the file are correct, press **Control Z** followed by Quit if the changes are not to be saved, or Exit if the changes are to be saved. Type **RANGE # #** to indicate which banks to show on the supervisor's action report.

14.11.3 At the $ prompt, type **Process** to start the processing of the background counts. The program proceeds to automatically process the background data one detector at a time.

14.12 Reviewing Weekly Background Report

14.12.1 After the data is processed, a Supervisor's Action Report will be printed. Review the printout for any out of control conditions. Contact Group Leader or designee immediately for out of control conditions. Detector should be logged out for isotopes that have a high background.

NOTE: Refer to GL-RAD-I-012, Managing Statistical Data in the Radiochemistry Laboratory, for guidance on locking out detectors.

14.12.2 Update detector status board.

15.0 OPERATING PROCEDURE

15.1 Sample Counting (Alpha Analyst detectors)

15.1.1 From the AMS Procedure window, select **Displays** then **Chamber Status**; ensure detectors are free for use.

15.1.2 Open the door of the sample chamber. Carefully remove any sample that is in the chamber with a pair of tweezers and place it in a storage container.

15.1.3 Using a pair of tweezers, carefully position the next sample that is to be counted on the sample shelf, taking care to center the sample beneath the detector face and ensure the sample shelf is in the proper location.

15.1.4 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.

15.1.5 Close the chamber doors.

15.1.6 From the AMS Procedure window, select **Count** then **Samples**.

15.1.7 Enter a list of the chambers that you are starting and click **OK** or press **Enter**.

15.1.8 A window will appear indicating all detectors have started. Click **OK**.

15.1.9 The detectors will automatically evacuate air from the chamber and apply a detector bias. The system starts data acquisition on all alpha counters. The default count time is set to four (4) hours, but can be changed during step 15.3.4 if necessary.

15.1.10 If data acquisition is to be started on model 7401 detectors at the same time, proceed to section 15.2.

15.1.11 If only Alpha Analyst detectors are to be started, return to the DECterm window before proceeding further.

15.1.12 From the DECterm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting System Main Menu is displayed, proceed with 15.1.13. If the Sample Counting System Main Menu is not displayed, consult the Group Leader or their designee.

15.1.13 Select **1) Sample Counting** to access the Sample Counting Menu.

15.1.14 Select **1) Count a New Sample** to access the Alpha Counting Menu.

15.1.15 Select **1) Samples**.

15.1.16 At the "Use which detector bank to count (RETURN to end)": prompt, press **Return**.

15.1.17 Enter sample information for each sample in accordance with section 15.3.

15.2 Sample Counting (model 7401 detectors)

15.2.1 From the DECterm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting Main Menu is displayed, proceed with 15.2.2. If the Sample Counting System Main Menu is not displayed, consult the Group Leader or their designee.

15.2.2 Select **1) Sample Counting** to access the Sample Counting Menu.

15.2.3 From Sample Counting Menu, Select **2) List Status of Detectors**; ensure detectors are free for use. Press **Return** to exit.

15.2.4 Ensure that there are no jobs active which use the alpha spectrometer(s) that is to be loaded. Ensure the bias supply to the detector is off, and then vent the chambers in accordance with section 15.5.

15.2.5 Open the door of the sample chamber. Carefully remove any sample that is in the chamber with a pair of tweezers and place it in a proper storage container.

15.2.6 Using a pair of tweezers, carefully position the next sample that is to be counted on the sample shelf, taking care to center the sample beneath the detector face and ensure the sample shelf is in the proper location.

15.2.7 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.

15.2.8 Close the chamber doors and start evacuation of the chambers in accordance with section 15.5.

15.2.9 After normal operating vacuum is achieved, turn on detector bias.

15.2.10 Before starting acquisition, verify the proper operating bias.

15.2.11 Select **1) Start a New Count** to access the Alpha Counting Menu.

15.2.12 Select **1) Samples**.

15.2.13 Enter the bank of detectors to acquisition on (or press **Return** when all needed banks are on) and the sample count time. Repeat for each bank as needed.

15.2.14 Enter sample information for each sample in accordance with section 15.3.

15.3 Entering Sample Information

15.3.1 Enter the Batch ID (or press **Return** when all count data has been entered and proceed to 15.3.6). The system will gather batch information from AlphaLIMS if this is the first time the batch number has been entered. If the system appears to hang while "Getting batch information from AlphaLIMS" is displayed, type **Control-Y** to skip to the next step. (In this case, no default information will be provided.)

15.3.2 Enter/verify appropriate batch parameters. Press **PF1** to accept the batch parameters and exit the Parameter Editor.

15.3.3 Enter the detector number containing the sample. If all samples for the batch have been entered, type **P** (if the batch is ready to proceed to data review) or C (if batch is remaining in the count room) and press **RETURN**. If there are more batches to count, continue with section 15.3.1. If no other batches are to be started press **RETURN** and go to section 15.3.6.

15.3.4 The system then enters the Parameter Editor. Enter/verify the displayed information for the sample.

15.3.5 Press **PF1** to exit the Parameter Editor. Continue with 15.3.3 for the next sample.

15.3.6 Monitor detectors for high count rates. If the count rate exceeds 100 counts per minute (across the entire spectrum), turn off the bias, vent the chamber, remove the sample from the chamber, and contact the Group Leader (or designee) immediately. A one-hour background count may be necessary to confirm that the detector was not contaminated.

15.3.7 When sample counts are finished, each sample may be processed automatically. If spectrum data needs to be manually processed, proceed with section 15.4.

15.4 Processing Sample Data via AlphaGEL

15.4.1 This section assumes the operator has a general understanding of the Microsoft Windows operating environment.

15.4.1.1 AlphaGEL is a custom software package developed exclusively for GEL Laboratories, which runs from a PC.

15.4.1.1.1 Start the client program from the Windows start menu or the desktop icon.

15.4.1.1.2 If necessary, click "Network", then "Connect" to connect to the AlphaGEL server. Optionally double-click on the red network status indicator. Enter Username and Password if necessary.

15.4.2 Enter the batch number of the samples to process in the main box of the "Processing" frame of the "AlphaGEL Remote Connection-Client" window and click "View Batch Info" or press **Enter**.

15.4.3 Verify all sample information in the "Batch Information" window.

15.4.3.1 Sample information can be corrected by either double-clicking a cell or selecting a cell and pressing Enter.

15.4.3.2 Double-clicking the column header can change an entire column. This will cause all values in the column to be the same.

15.4.3.3 Sample information changes are not saved until "Confirm All Changes" is clicked.

15.4.3.4 If changes are to be discarded, clicking "Tools", "Revert to Saved" will restore the information to the last saved state.

15.4.3.5 Verify that the radioactive standards are correct by clicking "Standard" and reviewing the "Standards Information" window.

15.4.4 Mark the samples from the batch to process by using the left-hand column. "X" indicates that the sample will be processed. All samples are marked for processing when the batch is first opened.

15.4.5 Verify that the "Client Processing Options" are correct. If any changes to these needs to be made, consult with the Group Leader or their designee.

15.4.6 Click "Process this Batch" to begin processing the marked samples.

15.5 Vacuum pump operation for model 7401 detectors

15.5.1 Evacuation of Air from Alpha Spectrometer model 7401

15.5.1.1 Ensure that the vacuum manifold control is set to the **"pump down manifold"** position.

15.5.1.2 Place the **"pump/vent"** valve/switch on the spectrometer in the **"pump" and locked** position.

15.5.1.3 Repeat 15.5.1.2 for each chamber in the bank.

15.5.1.4 Monitor the pump down manifold vacuum gauge until the indicator is below 10 millimeters of mercury.

15.5.1.5 Place the vacuum manifold control in the **"high vacuum manifold"** position. The detectors are now at normal operating vacuum.

15.5.2 Venting Alpha Spectrometer model 7401 to Atmosphere

15.5.2.1 Ensure that the vacuum manifold control is set to the **"pump down manifold"** position.

15.5.2.2 Place the **"pump/vent"** valve/switch on the spectrometer in the **"vent"** position.

15.5.2.3 Repeat 15.5.2.2 for each chamber in the bank.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-010 Counting Room Instrument Maintenance.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Refer to GL-RAD-D-003 Data Review, Validation, and Data Package Assembly.

18.0 POLLUTION/CONTAMINATION

Not applicable.

19.0 DATA RECORDING, CALCULATIONS, AND REDUCTION METHODS

Data recording, calculation, and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data might require instrument maintenance, re-analysis, using a new spike mix, or a more complex set of actions. When trouble-shooting measures fail to bring an analytical process or data into control, a Data Exception Report (DER) and/or corrective action should be initiated in accordance with AlphaLIMS Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, GL-QS-E-004 and/or Conducting Corrective/Preventive Action and Identifying Opportunities for Improvement, GL-QS-E-002.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting the instrument is a function of analyst experience. In-house service is obtained from GEL's Group Leader or other qualified personnel. If vendor assistance is needed, then the appropriate vendor is contacted. Maintenance logbooks are kept for each instrument and contain entries for both routine and non-routine maintenance procedures.

22.0 RECORDS MANAGEMENT

22.1 Each sample analysis that is performed is documented in the instrument run log in accordance with GL-LB-E-009 Run Logs.

22.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival in accordance with GL-RAD-D-003 Data Review, Validation, and Data Package Assembly.

22.3 Instrument maintenance is recorded in accordance with GL-LB-E-008, Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices.

23.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Refer to GL-LB-G-001 Laboratory Waste Management Plan.

24.0 REFERENCES

24.1 1990 Annual Book of ASTM Standards, Volume 12.02, E181.

24.2 Canberra Model 7401 Alpha Spectrometer Operations Manual.

24.3 U.S. Department of Energy Quality Systems for Analytical Services (DOE QSAS), Revision 2.8, January 2012

24.4 Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP), Chapter 18, July 2004

24.5 American Society for Testing and Materials (ASTM), ASTM C 1128-01(2008) and C1159-03 (2003).

24.6 National Institute of Science and Technology (NIST), Technical Note 1297, 1994.

25.0 HISTORY

Revision 14: Updated to comply with current process as part of annual review.
Revision 13: Added reference note for the preparation of calibration sources for alphaspectrometer. Removed guidelines for the preparation and recertification of rare-earth fluoride efficiency sources for alpha spectrometry.
Revision 12: Added section for processing samples via AlphaGEL.
Revision 11: Annual review: Updated SOP throughout.

APPENDIX 1: TROUBLESHOOTING

Symptom	Possible Cause	Solution
Vacuum light on Alpha Analyst pair continuously blinks (beyond normal pump down time)	Vacuum pump oil low	Check the oil level on the vacuum pump and fill if necessary.
	Debris on vacuum seal of door	From the AMS Procedure window, select **Count** then **Pause Acquire**. Select **AA** then **Vent Chambers**. Open both chambers & clean the seal with a dry lint free cloth. Close the chambers. From the AMS Procedure window, select **AA** then **Pump Chambers**. Select **Count** then **Unpause Acquire**.
Acquisition of Alpha Analyst detector paused	Chamber was manually paused	From the AMS Procedure window, select **Count** then **Unpause Acquire**. Enter the detector to unpause.
	Vacuum leak to chamber	See "Vacuum light on Alpha Analyst pair continuously blinks" for possible solutions.
	Electronic fault	Check to see if the "fault" light on the detector is on, if so, abort acquisition by selecting **Count** then **Abort Acquire** from the AMS Procedure window for the effected chambers. Restart the count in accordance with section 14.1, 14.5, 14.9, or 15.1 as appropriate.
Buttons and/or switches on model 7401 detector do not appear to be functioning	Multiple causes	Reset chamber by simultaneously pressing RESET and DIGIT SELECT and move the INC/DEC switch to INC. Release the buttons & switch when the display goes blank.
Apparent poor vacuum (7401 only) indicated by display (above 800 µm Hg) or vacuum gauge	Vacuum sensor of chamber is bad	If the majority of other detectors on the same vacuum pump indicate a proper vacuum, the vacuum sensor in the detector is likely malfunctioning. This will not hinder detector operation.
	Vacuum pump oil low	Check the oil level on the vacuum pump and fill if necessary.
	Debris on vacuum seal of door	Attempt to isolate the bank where the poor seal is by putting manifold controls to hold position for each bank on the same pump to see if the vacuum shows improvement. Once a specific bank is isolated, use the chamber vacuum controls to further isolate the individual chamber. Once a single chamber is isolated as the cause, open the chamber & clean the seal with a dry lint free cloth. Restart the count of the affected bank.

	Manifold valve leaking	Attempt to isolate the bank where the leak is by putting manifold controls to hold position for each bank on the same pump to see if the vacuum shows improvement. Once a specific bank is isolated, check that the seal of the manifold valve is tight. Remove the valve lever by loosening the screw under the larger end & tighten the nut around the manifold valve. Be sure the nut is not so tight that the valve cannot be turned. Replace the valve lever.
	Leaking or cracked vacuum hose	Inspect hoses for loose connections or cracks. Replace cracked hose if necessary. If seal is loose, tighten hose clamp and/or add vacuum grease.
	Cracked or worn vacuum connection inside chamber	This problem involves working close to the electronics inside a 7401 detector. Consult the Group Leader (or designee) regarding repair of this condition.
Vacuum control knob loose (7401 only)	Alan screw loose	Using an alan wrench, tighten the two screws inside the knob.
DECterm window is not displayed	Window closed or computer was restarted	Open a new DECterm window from the Session Manager toolbar by selecting **Applications** then **DECterm**.
AMS Procedure window is not displayed	Window closed or computer was restarted	Open a new AMS workspace from the Spectroscopy Assistant window by selecting **File** then **Open Workspace**. Select the file **HUME.WSP** then click **OK**.
Spectroscopy Assistant window is not displayed	Window closed or computer was restarted	Open a new Spectroscopy Assistant window from the Session Manager toolbar by selecting **Applications** then **AMS Spec. Assistant**.